

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12667-3 (1989): Methods of chemical analysis of chromite sand, Part 3: Determination of chromium [MTD 13: Ores and Raw Materials]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



Indian Standard

**CHROMITE SAND FOR FOUNDRIES —
METHODS OF CHEMICAL ANALYSIS**

PART 3 DETERMINATION OF CHROMIUM

भारतीय मानक

फाउंट्रियों के लिए क्रोमाइट सैंड — रासायनिक विश्लेषण

भाग 3 क्रोमियम ज्ञात करना

UDC 666·762·43 : 543 [546·76] : 621·742·42

© BIS 1989

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Part 3) was adopted by the Bureau of Indian Standards on 29 March 1989, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

IS 6788 : 1973 'Specification for chromite sand for foundries' covers the requirements for the various constituents. As chromite sand is widely used in steel/iron castings, a need was felt to prepare the standard on methods of test based on the requirements of IS 6788 : 1973.

This standard has been prepared in different parts. This part (Part 3) covers the chemical analysis of chromium, and other parts are:

Part 1 Determination of silica

Part 2 Determination of iron

Part 4 Determination of calcium

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***CHROMITE SAND FOR FOUNDRIES —
METHODS OF CHEMICAL ANALYSIS****PART 3 DETERMINATION OF CHROMIUM****1 SCOPE**

1.1 This standard (Part 3) covers a method for determination of chromium (as Cr_2O_3) in chromite sand for foundries.

2 REFERENCE

IS 1811 : 1984 Methods of sampling foundry sands (*first revision*)

3 SAMPLING

3.1 The sample shall be drawn by the method prescribed in IS 1811 : 1984.

3.2 Pulverize about 10 g of sample in an agate mortar to pass through 75 μm IS test sieve. Dry at 105 to 110°C for 2 hours.

4 GENERAL**4.1 Use of Filter Paper**

In this method, only relative number of Whatman filter paper has been prescribed since they are commonly used. However, any other suitable brand of filter paper of corresponding porosity and equivalent quality may also be used.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

6 DETERMINATION OF CHROMIUM BY TITRIMETRIC METHOD**6.1 Out line of the Method**

Sample is fused with sodium peroxide and the mass extracted with water. Chromium is oxidized to dichromate and excess ferrous ammonium sulphate is added which is titrated with standard potassium dichromate solution.

6.2 Reagents

6.2.1 Sodium Peroxide, granular.

6.2.2 Dilute Hydrochloric Acid, 1 : 1 (*v/v*).

6.2.3 Ammonium Chloride Solution, 10 percent (*m/v*).

6.2.4 Ammonium Hydroxide, rd = 0.90.

6.2.5 Dilute Sulphuric Acid, 1 : 1 (*v/v*).

6.2.6 Potassium Permanganate Solution, 2 percent (*m/v*).

6.2.7 Ammonium Persulphate Solution, 25 percent (*m/v*).

6.2.8 Sodium Nitrite, 0.5 percent (*m/v*).

6.2.9 Ferrous Ammonium Sulphate Solution (0.1 N Approx)

Transfer 39.21 g of ferrous ammonium sulphate crystals to a 1 litre volumetric flask and pour in around 200 ml water. Shake well for the crystals to dissolve. Add 100 ml dilute sulphuric acid (1 : 1) and make up to mark with water. Shake well and preserve in a well-stoppered bottle.

6.2.10 Sulphuric-Phosphoric Acid Mixture

Add 150 ml of concentrated sulphuric acid (rd = 1.84) to 700 ml water slowly with continuous stirring. Add to this, 150 ml syrupy phosphoric acid (85 percent). Destroy any oxidizable impurity by adding potassium permanganate solution (0.1 N) dropwise till pink colour persists. Cool the solution.

6.2.11 Sodium Diphenylamine Sulphonate Indicator Solution

Dissolve 0.32 g of barium diphenylamine sulphonate in 100 ml hot water. Add 0.5 g of sodium sulphate, stir and filter off the precipitate of barium sulphate. Store in a dark coloured bottle.

6.2.12 Standard Potassium Dichromate Solution (0.1 N)

Dry pure crystals of potassium dichromate (AR quality) in an air oven at 105 to 110°C for an hour. Cool in a desiccator and transfer carefully 4.903 g through a funnel to a 1-litre volumetric flask using water at room temperature. Wash the funnel and dissolve the salt by shaking. Make up to mark and mix thoroughly. Preserve the solution in a well-stoppered bottle.

6.3 Procedure

6.3.1 Weigh 0.500 g of sample (see 3.2) into a 30 ml nickel crucible. Add 4 g of sodium peroxide and mix thoroughly by means of a thin glass rod. Remove any sample powder adhering to the rod by stirring about 1 g sodium peroxide with it. Gently heat the covered crucible to dull red heat and swirl the melt to ensure rapid completion of reaction. Keep in molten state for around ten minutes. Allow to cool and place the crucible with contents in a 400-ml beaker containing around 50 ml water. Cover the beaker with a watch glass and allow the solidified mass to react with water. Wash the crucible and lid into the beaker and remove these out. Dilute to around 100 ml and continue boiling for ten minutes. Cool and filter through Whatman filter paper 540 with pulp, washing the residue well with hot water. Keep the filtrate (A) aside.

6.3.2 Acidify the filtrate with dilute sulphuric acid, boil, cool and make up to 250 ml in a volumetric flask.

6.3.3 Take a suitable aliquot containing about 50 mg of chromium in a beaker, add 15 ml of (0.8 percent) silver nitrate solution, 2 drops of (2 percent) potassium permanganate solution, and 10 ml of (25 percent) ammonium persulphate solution, boil the solution for 10 minutes to oxidize chromium and manganese and to destroy excess of persulphate. Add 0.5 percent sodium nitrite solution drop by drop to boiling solution till the reduction of potassium permanganate solution and subsequent decomposition of any precipitated manganese dioxide and then add 1 to 2 drops in excess. Add 1 g of urea and stir to dissolve. Cool the solution.

Add 50 ml of 0.1 N ferrous ammonium sulphate solution. Shake up and add 115 ml of sulphuric-phosphoric acid mixture. Dilute to around 200 ml with water, add three drops of sodium diphenylamine sulphonate indicator solution and titrate it slowly with standard 0.1 N potassium dichromate solution stirring constantly until the green colour begins to darken. Continue the titration slowly till a drop of dichromate solution gives a permanent violet-blue coloration. Note the titre value.

6.3.4 Blank

Take 50 ml of 0.1 N ferrous ammonium sulphate solution in a 250 ml conical flask. Add acid mixture and dilute to 20 ml. Add few drops of sodium diphenylamine sulphonate indicator solution and titrate with standard 0.1 N potassium dichromate solution as above to a permanent violet-blue end point.

6.4 Calculation

Chromium (as Cr_2O_3), percent = $\frac{(A - B) \times 0.002534}{C} \times 100$

where

A = volume in ml of standard potassium dichromate solution consumed for 50 ml ferrous ammonium sulphate (blank) solution,

B = volume in ml of standard potassium dichromate solution consumed for 50 ml ferrous ammonium sulphate with sample aliquot, and

C = mass in g of sample represented by the aliquot taken.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Revision of Indian Standards

Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent to BIS giving the following reference:

Doc : No. SMDC 35 (2877)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 331 01 31 331 13 75
Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054	36 24 99
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	{ 2 18 43 3 16 41
Southern : C.I.T. Campus, IV Cross Road, MADRAS 600113	{ 41 24 42 41 25 19 41 29 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	6 32 92 95
Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. PATNA. TRIVANDRUM.	